EFFECT OF IRON (Fe^{3+}) DOPED CALCIUM COPPER TITANATE OXIDE (CCTO)

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SUPERVISORS' DECLARATION

I hereby declare that I have checked the thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Applied Science (Honor) Material Technology.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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DEDICATION

I dedicate this thesis to my family especially to my parent whose who help me unconditionally to finish this thesis.

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Praise God for good health and wellbeing so that I could finish this thesis. I'm grateful to God for this opportunities and granting me successfully to finish this thesis.

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ABSTRACT

The $CaCu_3Ti_{4-x}Fe_xO_{12}$ (x = 0, 0.05 and 0.1) ceramics were synthesized by solid state reaction route and the microstructure and dielectric properties were studied. The X-Ray Diffraction (XRD) was used to analysis the lattice parameter and it was found that as the dopant contents increase the lattice parameter decrease. The microstructures were analyzed using Field Emission Scanning Electron Microscopy (FESEM) and it shows that as the dopant content increase the grain size increase. The dielectric measurement were performed at room temperature shows that with pure Calcium Copper Titanate (CCTO) doped with iron (Fe^{3+}), the dielectric permittivity (ε) at frequency 10 Hz decreases. Same goes with dielectric loss ($\tan \delta$), decrease when pure CCTO doped with iron (Fe^{3+}).

ABSTRAK

$CaCu_3Ti_{4-x}Fe_xO_{12}$ (x = 0, 0.05 dan 0.1) seramik telah disintesis melalui tindak balas dalam keadaan pepejal dan mikrostruktur dan sifat dielektrik telah dikaji. Pembelauan X-Ray (XRD) telah digunakan untuk menganalisis parameter kekisi dan didapati apabila kandungan pendopan meningkat maka parameter kekisi berkurangan. Mikrostruktur telah dianalisis dengan menggunakan Pelepasan Bidang Imbasan Mikroskop Electron (FESEM), dan telah menunjukkan bahawa apabila kandungan pendopan meningkat saiz butiran juga meningkat. Sementara itu pengukuran dielektrik telah dijalankan pada suhu bilik menunjukkan bahawa dengan Calcium Copper Titanate (CCTO) tulen didopkan dengan besi (Fe^{3+}), permittiviti dielektrik (ε) pada frekuensi 12 Hz berkurangan. Begitu juga dengan dielektrik loss (tan δ) menurun apabila CCTO tulen didopkan dengan besi (Fe^{3+}).

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LIST OF SYMBOLS

ε'	-	Dielectric permittivity
$\tan(\delta)$	-	Dielectric loss
λ	-	Wavelength
μ	-	micron (10^{-6})
ε	-	Dielectric constant
2θ	-	Bragg angle
^{o}C	-	Degree Celcius
Å	-	Angstrom (10^{-10})
ω	-	Angular velocity
g/mol	-	Molecular weight
A	-	Sample area
t	-	Sample thickness
ε_o	-	Permittivity of free space
a	-	Lattice parameter
Zi	-	Imaginary impedance
Zr	-	Real impedance

LIST OF ABBREVIATIONS

CCTO	-	Calcium copper titanium oxide
CCTFO	-	Calcium copper titanium iron oxide
Ca	-	Calcium
Cu	-	Copper
Ti	-	Titanium
Fe	-	Iron
O	-	Oxygen
IBLC	-	Internal barrier layer capacitance
EDLC	-	electrochemical double layer capacitance
FESEM	-	Field Emission Scanning Electron Microscopic
XRD	-	X-ray diffraction
PVA	-	Polyvinyl alcohol
Y	-	Ytterium

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Microelectronic can be defined as the area of technology that associated with and applied to realization of electronic systems which is extremely small electronic elements or parts. Basically, microelectronic is subfield of electronic devices, which are relates to the manufacture and study of components and electronic designs. Few years ago, the discovery of high permittivity in Calcium Copper Titanate (CCTO) by Subramanian *et al.*, (2000), CCTO has become an important and suitable ceramic material to replace commonly used high-dielectric materials. CCTO had high dielectric constant which is more than 10^4, and also has a good stability, beside, it also has low loss tangent. CCTO structure is a complex body centered cubic perovskite oxide having a very large value of dielectric and almost constant in temperature range 100-600 K. The structure of CCTO was derived from the cubic perovskite (ABO_3) by an octahedral tilt distortion which caused by mismatch of the size and the nature of A cations. Meanwhile, the TiO_6 octahedral tilt produce a structure where three quarter of the A sites have quarter planar coordination which are occupied with Cu ions and the remaining quarters of the sites are occupied by Ca atom which is have 12 fold coordination.

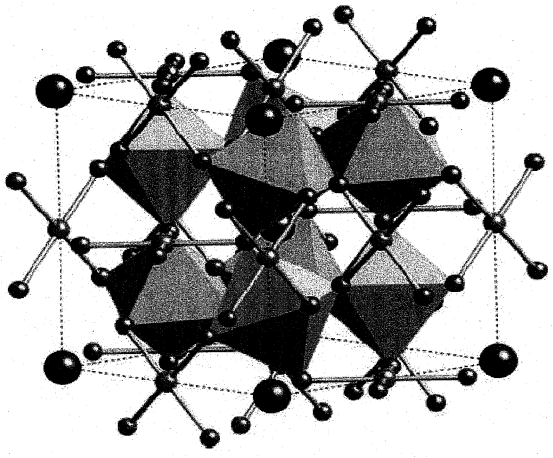

Figure 1.1: Structure of $CaCu_3Ti_4O_{12}$ which shown octahedral of TiO6, Cu atoms bonded to four oxygen atoms, and Ca atom without bond.

Source: Sunil Patra *et al.*, 2009

Mostly, materials with high dielectric constants are widely used in many applications in technology such as supercapacitor, capacitor, resonators and filters. Because of the high dielectric constant, CCTO is allowed smaller capacitive components, which are offering the opportunity to decrease the size of the electronic devices. With the ability to reduce the size of the electronic device, CCTO become the most important material in production of electronic devices.

Unfortunately, the origin of this giant high dielectric constant (high permittivity) are still unknown and not clear and intensively discussed in literature. However, a researcher like Homes et al., and Ramirez et al., explained the temperature behavior of permittivity in CCTO single crystals. They suggest that the polarization of mechanism involving displacement of Ti^{4+} ions from the center of symmetry within the octahedral sites. However, until today there is no experimental and research evidence to support this theory. Meanwhile, Li et al. and Whangbo and Subramanian, argued that the factor that caused high permittivity in CCTO was the present of intrinsic twin boundaries or planar defects. But nowadays, most of researcher accept the theory that the association of internal barrier layer capacitance (IBLC) with present of high dielectric constant of CCTO. Even though CCTO has high dielectric constant, but it also has high dielectric loss.

1.2 PROBLEM STATEMENT

Over the year, high dielectric constant are found in ferroelectric materials, but unfortunately calcium copper titanate (CCTO) is not a ferroelectric material and therefore, further studies about CCTO ceramics is ongoing. Most of the researchers are still had argument about the origin of high dielectric constant in CCTO ceramics. However, recently studied was found out that even though CCTO has high dielectric constant, it also had high dielectric loss. Fabrication of CCTO with certain element such as Yb, La and Fe will alter the dielectric properties and morphology of the CCTO. In this experiment, CCTO will doped with Fe^{3+} in order to understand and learn the factor that cause high dielectric loss in CCTO.

1.3 OBJECTIVES OF RESEARCH

Objectives of this research are:

1. To synthesis calcium copper titanate (CCTO) doped with Fe^{3+} with composition of $x = 0.00$, $x = 0.05$ and $x = 0.1$ through solid state method.
2. To study the dielectric loss and dielectric permittivity of calcium copper titanate (CCTO) doped with Fe^{3+} using Potentiostat.
3. To investigate the morphology of calcium copper titanate (CCTO) doped with Fe^{3+} via FESEM.

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